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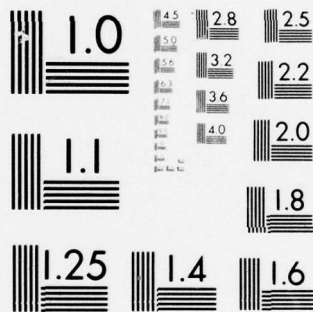
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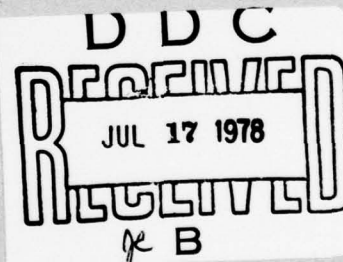
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TECHNICAL REPORT ARLCD-TR-77047

CONTINUOUS MONITORING OF PINK WATER
FROM CARBON ADSORPTION PROCESS

MILTON ROTH
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APRIL 1978



US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
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<p>An adaptation of a commercially available photometric analyzer has been evaluated for its suitability in measuring and monitoring pink water (TNT, RDX, and other nitrobodyes) on a continuous, real-time basis. A study was made to identify the interferences in the monitoring system and means for their elimination were developed. Calibration curves were obtained for TNT, RDX, and admixtures. The effect of light on the stability of the solutions was shown to be inconsequential in causing drift in meter reading.</p>		

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20. Abstract (con't):

Although both TNT and RDX contribute to the absorption at the measuring wavelength, the latter can be measured independently because it precedes the TNT in eluting from the carbon column. The TNT appears only after the RDX concentration has risen to a level that is a signal for replacement of the carbon. The complete design and operation of the monitoring system is shown.

With the existing system, at appropriate wavelengths, the sensitivity is suitable for measuring as little as 0.1 ppm of TNT or 1 ppm of RDX. Modification of the system to increase the pathlength and/or to change the measuring wavelength could produce further increases in sensitivity.

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FOREWORD

The work described in this report was conducted under Task 19, "Methods and Equipment to Measure, Monitor, and Control Pollutants" as part of MM&T Project 4114, "Development of Methods to Minimize Environmental Contamination".

The first phase of the work, done at Iowa AAP, was the evaluation of a DuPont 400 photometric analyzer, using ultraviolet absorption for measuring TNT concentration in the eluate from the carbon adsorption process for pink water. The second phase, initiated at Iowa AAP and completed at ARRADCOM, consisted of adapting the analyzer to on-line use for continuous monitoring and control of TNT and RDX coming from the carbon adsorption process.

The second phase also demonstrated that TNT does not decompose during exposure to radiation from the measuring or reference wavelengths used in the analyzer. Under the conditions used, TNT concentrations of 0.1 ppm and up can be monitored on a continuous basis. Approaches for even greater sensitivity are also described in this report.

This project was accomplished as part of the US Army manufacturing technology program. The primary objective of this program is to develop, on a timely basis, manufacturing processes, techniques, and equipment for use in production of Army materiel.



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INTRODUCTION

Upon exposure to light, aqueous solutions of TNT change from colorless to shades of pink or red. Untreated wastewaters from manufacturing, bomb-loading, assembling, and packing of TNT contaminate receiving waters, and the color which develops provides visible evidence of pollution. Although there are no official limits on the concentration of TNT which may be discharged into receiving waters, 0.5 ppm has been suggested as a guideline.

A number of methods have been developed that are sufficiently sensitive for the analysis of TNT in wastewater. Kay (ref. 1) and Elvove (ref. 2) have developed colorimetric methods which are mainly suitable for qualitative tests. Two colorimetric methods used for quantitative TNT analyses are: 1) the Ruchhoft-Meckler method, which is based on sodium sulfite-sodium hydroxide for color development with a measuring wavelength of 505 nm and 2) the Silas-Mason method, which is based on 2-diethylaminoethanol with a measuring wavelength of 425 nm. The reagent, diethylaminoethanol, was first used by Goldman (ref. 3) to distinguish between TNT, which gives a violet color, and tetryl, which gives a red. Ethanolamines give no color reaction with DNT, (ref. 4), but if an alkali is added, a blue-green color develops. Both of these colorimetric methods are inadequate for continuous monitoring.

For continuous on-line monitoring, spectrophotometry offers one of the best approaches and Iowa AAP was directed to evaluate a photometric analyzer for linearity, sensitivity, specificity, response time, stability, reproducibility, frequency of calibration, and zero drift. Concurrently, Iowa AAP developed a sampling system (ref. 5) which included installation of 1) air venting valves on each carbon column to eliminate bubbles which were a source of errors; 2) in-line filters between the instrument and the columns to remove carbon fines and other suspended matter; 3) a 3-way valve arrangement which allows the effluent or distilled water or standard solutions to pass through the sample cell, and 4) a control loop to change columns as they become depleted.

The work was completed at ARRADCOM, in order to use ARRADCOM facilities to establish a calibration method, to demonstrate the stability of TNT solutions when exposed to the measuring and reference radiation, and to provide an orderly procedure for on-line monitoring. In addition, analytical methods for TNT were surveyed.

CARBON ADSORPTION PROCESS

Column Operation

The carbon adsorption process for treatment of TNT wastewaters is shown in figure 1. The wastewater passes through columns packed with activated carbon (Filtrisorb 300). From the column outlet, a sample stream passes through a filter, which removes particulates, and into the monitor where TNT or another nitrobody is measured. The measurement is displayed on a meter and/or strip chart recorder. The recorder can be preset at a point corresponding to a specific concentration of TNT which, when exceeded, activates a valve that switches the flow from the depleted column to a fresh one.

Monitor Design

The front and rear panels of the monitor are shown in figures 2 and 3. The front panel contains the electrical controls (fig. 2) while the rear panel contains the components of the monitor (fig. 3). The sample cell has a path length of 7 inches, but the compartment can accommodate up to a 9-inch cell and modifications can be made for even longer cells. The principle of operation is based on the absorption of uv light by the sample material. As shown in figure 4, radiation from the light source passes through the sample to a semi-transparent mirror where the beam is split so that half is directed through an optical filter at a wavelength that is suitable for measurement of the constituent of interest while the other half is directed through another filter at a wavelength which corrects for the background of the sample stream. Phototubes convert the transmitted energy to proportional electrical currents. The final output is a meter reading in accordance with Beer's Law.

Effect of Ultraviolet Radiation on TNT, RDX, and HMX Solution

Work at Iowa AAP (ref. 5) indicated that TNT solutions rapidly decompose on exposure to uv radiation resulting in a decreased absorption at the measuring wavelength. To determine whether the radiation within the analyzer really caused a change in absorbance, solutions containing 0.1 ppm and 2.5 ppm TNT in the sample cell were exposed for half an hour to uv radiation. A change of approximately 0.5 divisions in the meter reading, equivalent to about 0.025 ppm TNT, was found in each of duplicate tests. In addition, 1.00 ppm TNT in water was exposed to a similar wavelength on a Perkin Elmer spectrophotometer for 6 hours in a 5-cm cell and scanned every hour between 200-400 nm. The spectra were identical (superimposed) for each of the 6 hours (fig. 4), clearly indicating that TNT is stable under the test conditions. As a final check, solutions containing 0.5, 1.0, 2.5, 5.0 ppm of TNT, RDX, and HMX were stored for 30 days. Half of each

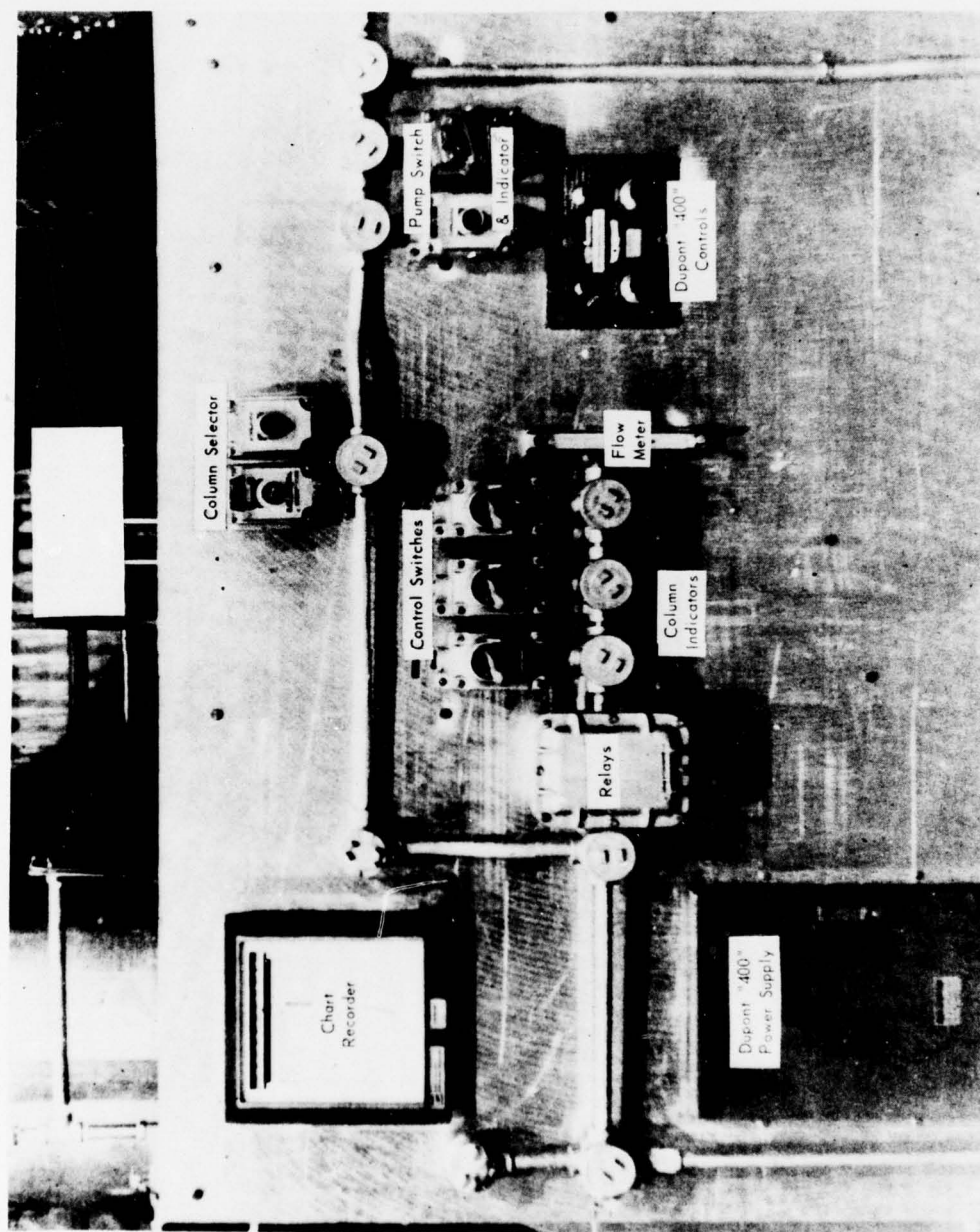


Figure 2. Nitrobody monitor, front panel

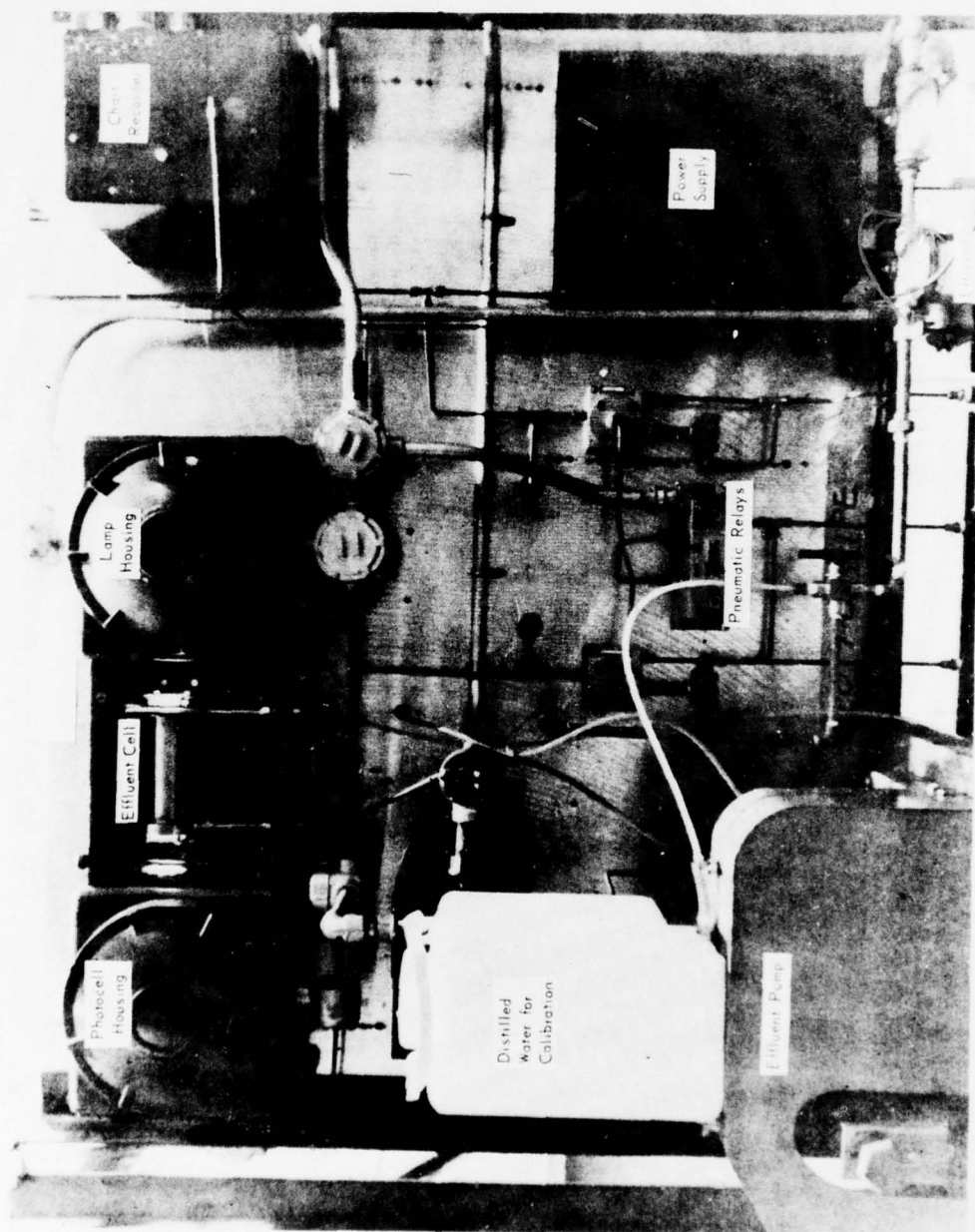


Figure 3. Nitrobody monitor, rear panel

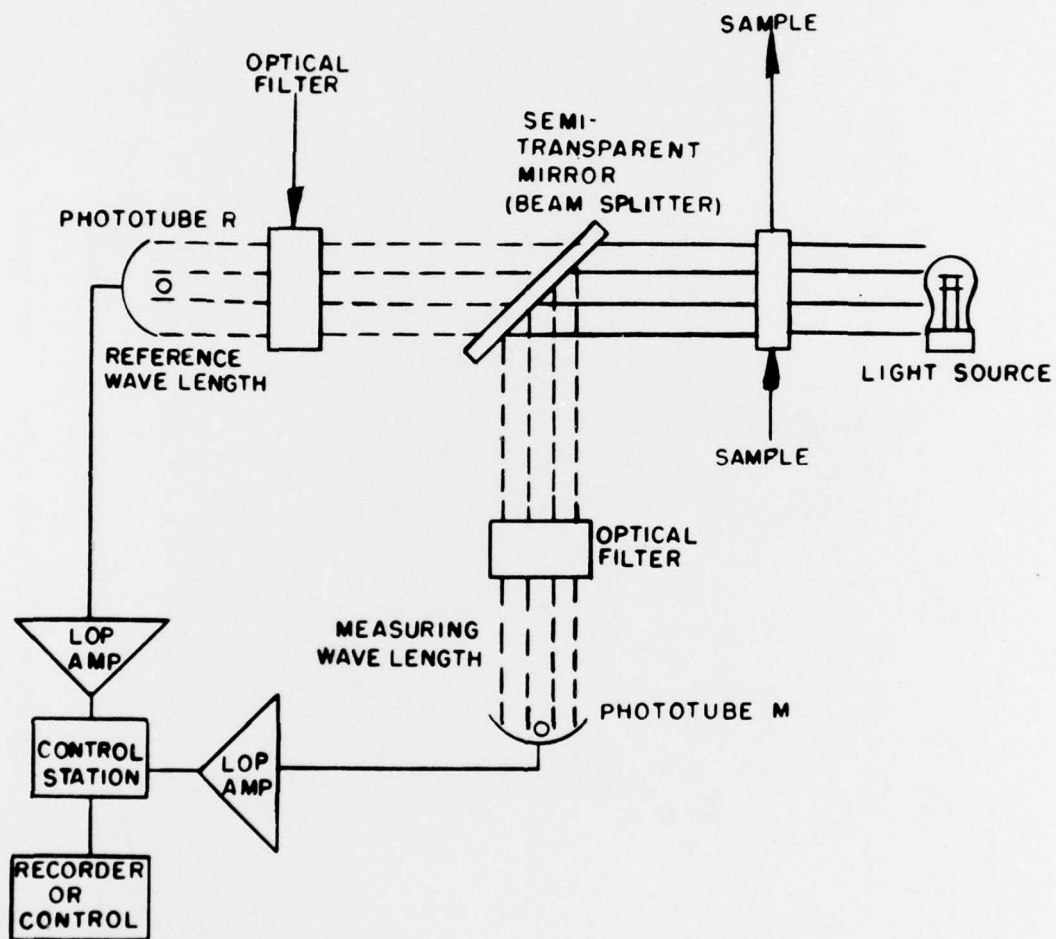


Figure 4. Principle of operation of photometric analyzer

solution was exposed to light. At weekly intervals, each solution was scanned between 200 and 400 nm. The data, tables 1 and 2, show a significant increase in absorbance in the exposed 2.5 ppm and 5.0 ppm TNT solutions at 280 and 365 nm (the measuring and reference wavelength) during the first week. During the second and third weeks, the 2.5 ppm solution remained constant followed by a further increase in the fourth week. The 5.0 ppm solution leveled off after the second week. No significant changes were observed in the unexposed solutions at either wavelength. RDX and HMX solutions appeared totally unaffected by exposure to light. The data for 2.5 and 5.0 ppm solutions of TNT are graphically shown in figures 6 and 7.

MONITORING PROCEDURES

Preparation of Standard Solutions

TNT

Weigh 100 milligrams (mg) of TNT to the nearest mg, transfer to a suitable container, and add 1500 ml of distilled water. Stir and heat the mixture until solution is complete. Cool to room temperature, transfer quantitatively to a 2-liter volumetric flask, and dilute to the mark. This stock solution contains 50 ppm of TNT. (All TNT solutions must be protected from light either by storing in a dark place or in an opaque container at $25^{\circ} \pm 5^{\circ}\text{C}$. Lower temperatures may cause separation of the TNT.) From the stock solution, take aliquots to prepare standard solutions corresponding to 0.1, 0.5, 1.0, 2.5 and 5.0 ppm of TNT.

RDX

Weigh 40 mg of RDX to the nearest mg, transfer to a suitable container, and add 1500 ml of distilled water. Stir and heat the mixture until solution is complete. Cool to room temperature, transfer to a 2-liter volumetric flask, and dilute to the mark. This stock solution contains 20 ppm of RDX. From the stock solution, prepare standard solutions corresponding to 1.0, 5.0, 10.0 and 20.0 ppm of RDX.

Calibration

Activate the DuPont 400 photometric analyzer as directed in the manufacturer's operating manual. Connect the inlet tube of the photometric cell to pass distilled water through the cell. When a constant meter reading is obtained, adjust the span control to set the meter at zero.

Table 1. Absorbance of TNT, RDX, and HMX at 280 and 365 nm
unexposed to light (5 cm cell)

Expl	ppm	<u>Days exposed to light</u>											
		0		8		15		22		30			
		280	365	280	365	280	365	280	365	280	365	280	365
TNT	0.5	0.00	0.00	0.02	0.00	0.00	0.00	0.08	0.00	0.06	0.00		
	1.0	0.04	0.00	0.08	0.00	0.06	0.00	0.08	0.00	0.10	0.00		
	2.5	0.22	0.02	0.21	0.01	0.16	0.03	0.18	0.01	0.23	0.02		
	5.0	0.15	0.02	0.40	0.02	0.34	0.00	0.40	0.01	0.44	0.04		
RDX	0.5	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.00		
	1.0	0.00	0.00	0.02	0.00	0.00	0.00	0.00	0.00	0.02	0.00		
	2.5	0.02	0.05	0.04	0.02	0.00	0.04	0.01	0.04	0.04	0.02		
	5.0	0.05	0.05	0.14	0.00	0.10	0.00	0.14	0.00	0.16	0.00		
HMX	0.5	0.00	0.00	0.02	0.00	0.00	0.00	0.00	0.00	0.01	0.00		
	1.0	0.00	0.02	0.02	0.00	0.00	0.00	0.00	0.00	0.02	0.00		
	2.5	0.02	0.04	0.04	0.03	0.02	0.06	0.01	0.04	0.04	0.01		
	5.0	0.04	0.00	0.06	0.00	0.00	0.00	0.03	0.00	0.06	0.00		

Table 2. Absorbance of TNT, RDX, and HMX at 280 and 365 nm
exposed to light (5 cm cell)

		<u>Days exposed to light</u>											
<u>Expl</u>	<u>ppm</u>	<u>0</u>		<u>8</u>		<u>15</u>		<u>22</u>		<u>30</u>			
		<u>280</u>	<u>365</u>	<u>280</u>	<u>365</u>	<u>280</u>	<u>365</u>	<u>280</u>	<u>365</u>	<u>280</u>	<u>365</u>	<u>280</u>	<u>365</u>
TNT	0.5	0.00	0.00	0.06	0.00	0.06	0.00	0.07	0.00	0.10	0.00		
	1.0	0.07	0.00	0.16	0.02	0.15	0.02	0.16	0.02	0.18	0.08		
	2.5	0.22	0.02	0.43	0.12	0.43	0.14	0.43	0.14	0.49	0.19		
	5.0	0.40	0.00	0.70	0.18	0.88	0.30	0.90	0.32	0.94	0.38		
RDX	0.5	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.04	0.00		
	1.0	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.00	0.06	0.00		
	2.5	0.02	0.05	0.06	0.02	0.00	0.04	0.06	0.02	0.08	0.01		
	5.0	0.14	0.00	0.18	0.00	0.15	0.00	0.18	0.00	0.18	0.02		
HMX	0.5	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.04	0.00		
	1.0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.06	0.00		
	2.5	0.02	0.04	0.04	0.02	0.02	0.04	0.43	0.04	0.08	0.00		
	5.0	0.02	0.00	0.06	0.00	0.04	0.00	0.04	0.00	0.08	0.00		

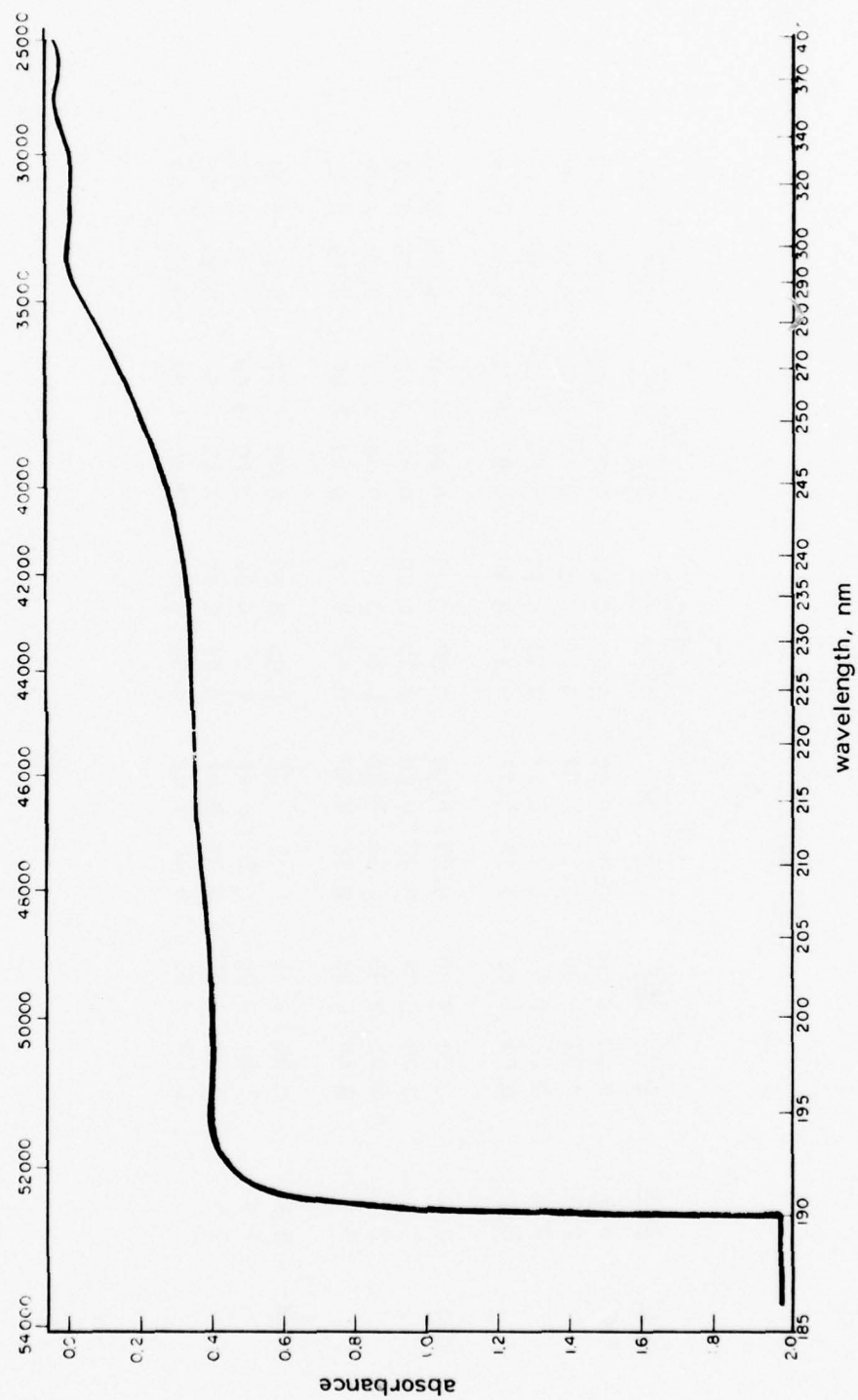


Figure 5. Absorbance of 1.0 ppm TNT after 6 hours exposure to ultraviolet radiation

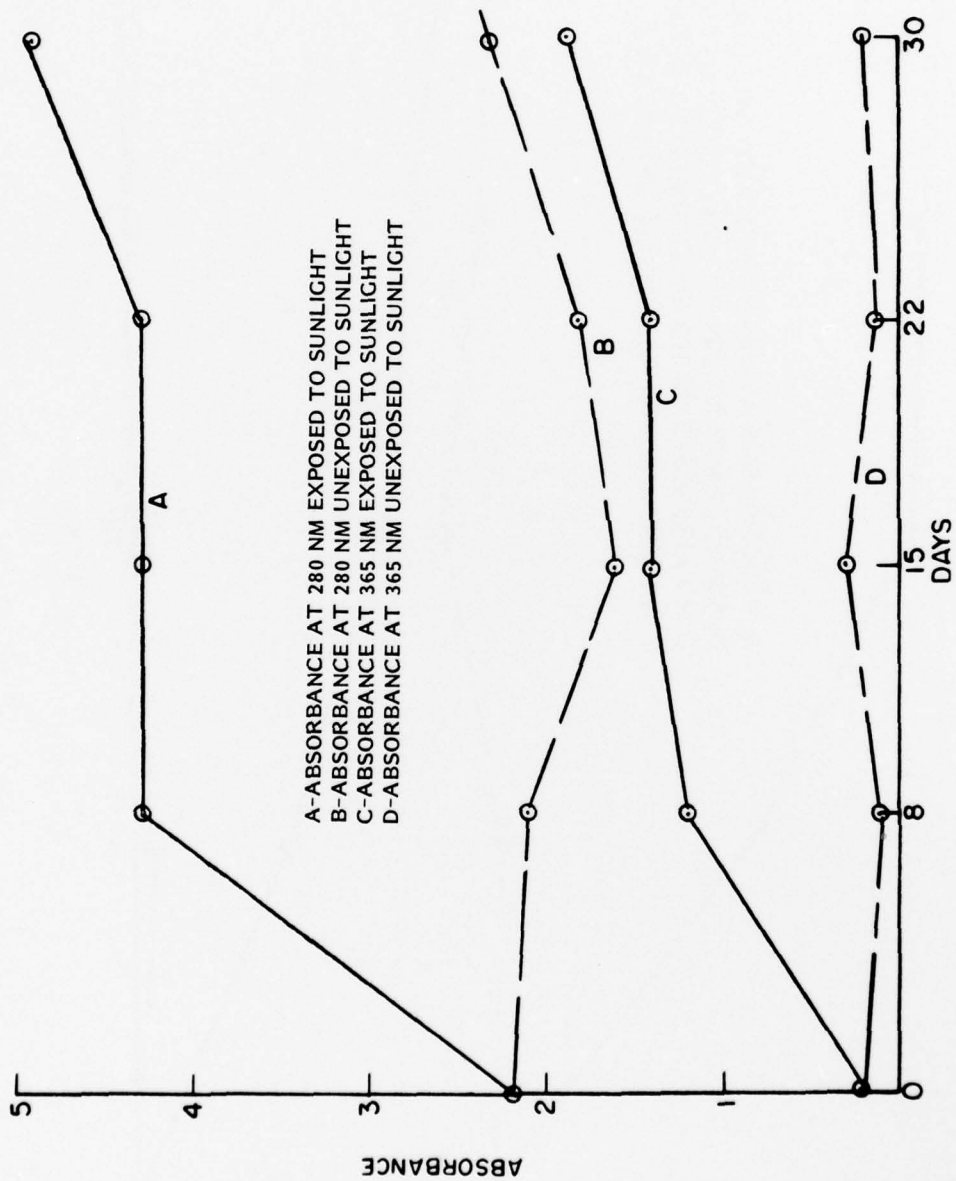


Figure 6. Absorbance of 2.5 ppm TNT solutions

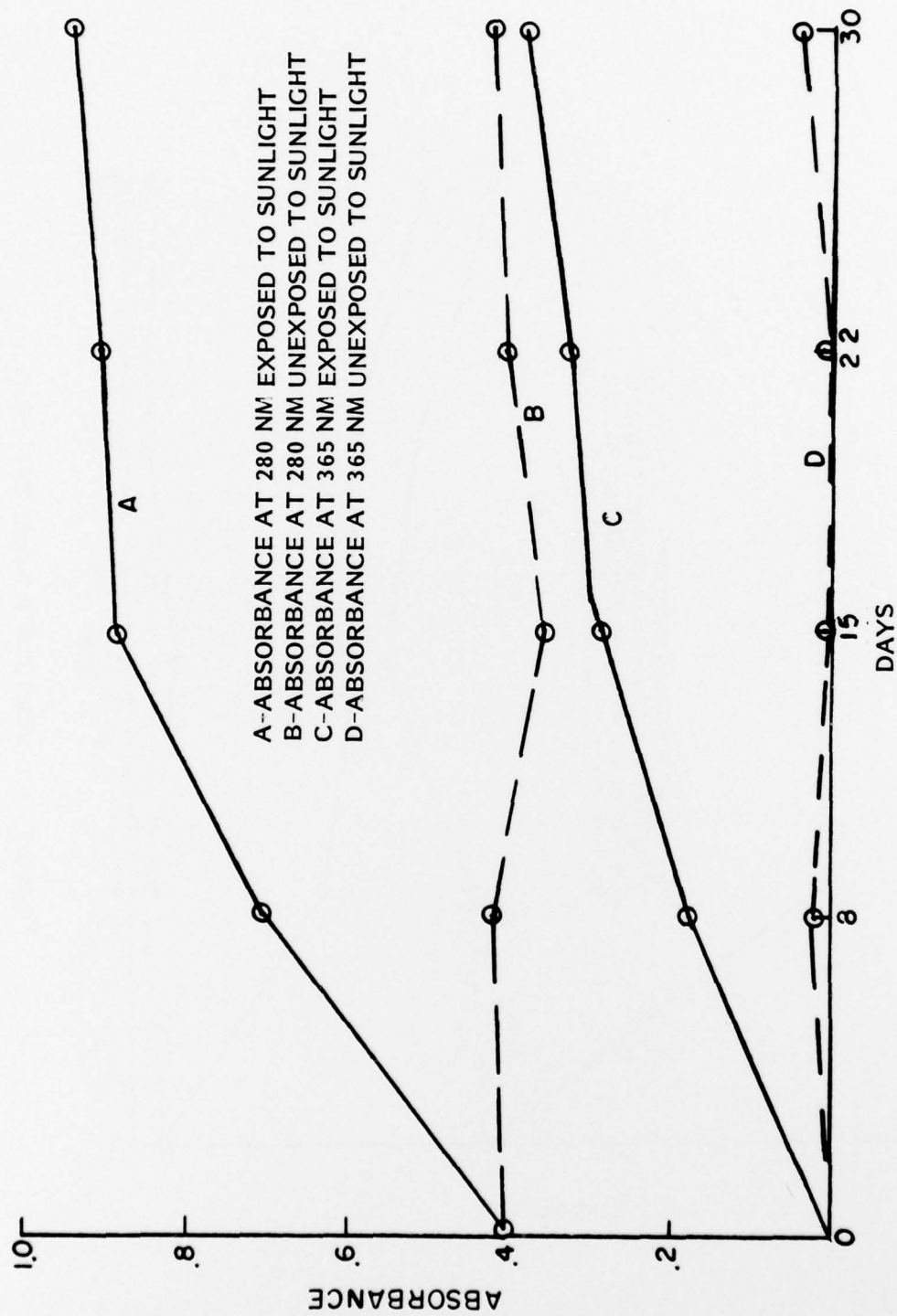


Figure 7. Absorbance of 5.0 ppm TNT solutions

In a similar manner, pass the 5 ppm TNT standard solution through the cell and adjust the span control to set the meter at 100. Without further adjustment of the span control, record the meter readings obtained by passing the other standard solutions (TNT and RDX) through the sample cell. From the readings obtained, plot calibration curves for TNT and RDX. Table 3 shows calibration data obtained and figure 8 shows the calibration curves plotted from these data.

Calibration must be corrected for absorption due to the carbon and the process water. To accomplish this, one liter of the process water is mixed with about 100 grams of the activated carbon used in the columns and allowed to stand for at least 2 hours. The mixture is then filtered, and the filtrate is passed through the DuPont analyzer. When the meter reading has stabilized, adjust the span so that the reading is zero. This zero correction should be made each time a new lot of carbon is used.

Reconnect the DuPont analyzer to the sample line from the carbon column and observe the reading produced by the effluent. If the effluent contains only TNT, use the TNT calibration curve. If both TNT and RDX are present, use the RDX calibration curve since this compound will be the first one to be eluted from the carbon column.

ANALYTICAL METHODS

During the course of the work at ARRADCOM, four colorimetric and five instrumental methods applicable to the determination of TNT content in wastewater were investigated. The four most useful methods (two colorimetric and two instrumental) are described below. Other methods are covered in references 6, 7, and 8.

Colorimetric Methods

Silas-Mason Method (ref. 9)

This method uses 2-diethylaminoethanol for color development and a 425-nm measuring wavelength. Sensitivity is better than 1.0 ppm with a 1.0-cm cell. The disadvantages of the method are that each sample must be heated exactly the same length of time at the same temperature in order to obtain reproducibility when converting to the reddish complex, and pH control must be maintained precisely.

Table 3. Calibration data for TNT and RDX

<u>TNT, ppm</u>	<u>Meter reading</u>
0.0	0.0
0.1	3.5
0.5	8.0
1.0	21.3
2.5	49.5
5.0	100.0

<u>RDX, ppm</u>	<u>Meter reading</u>
1.0	5.0
5.0	24.0
10.0	39.5
15.0	56.6

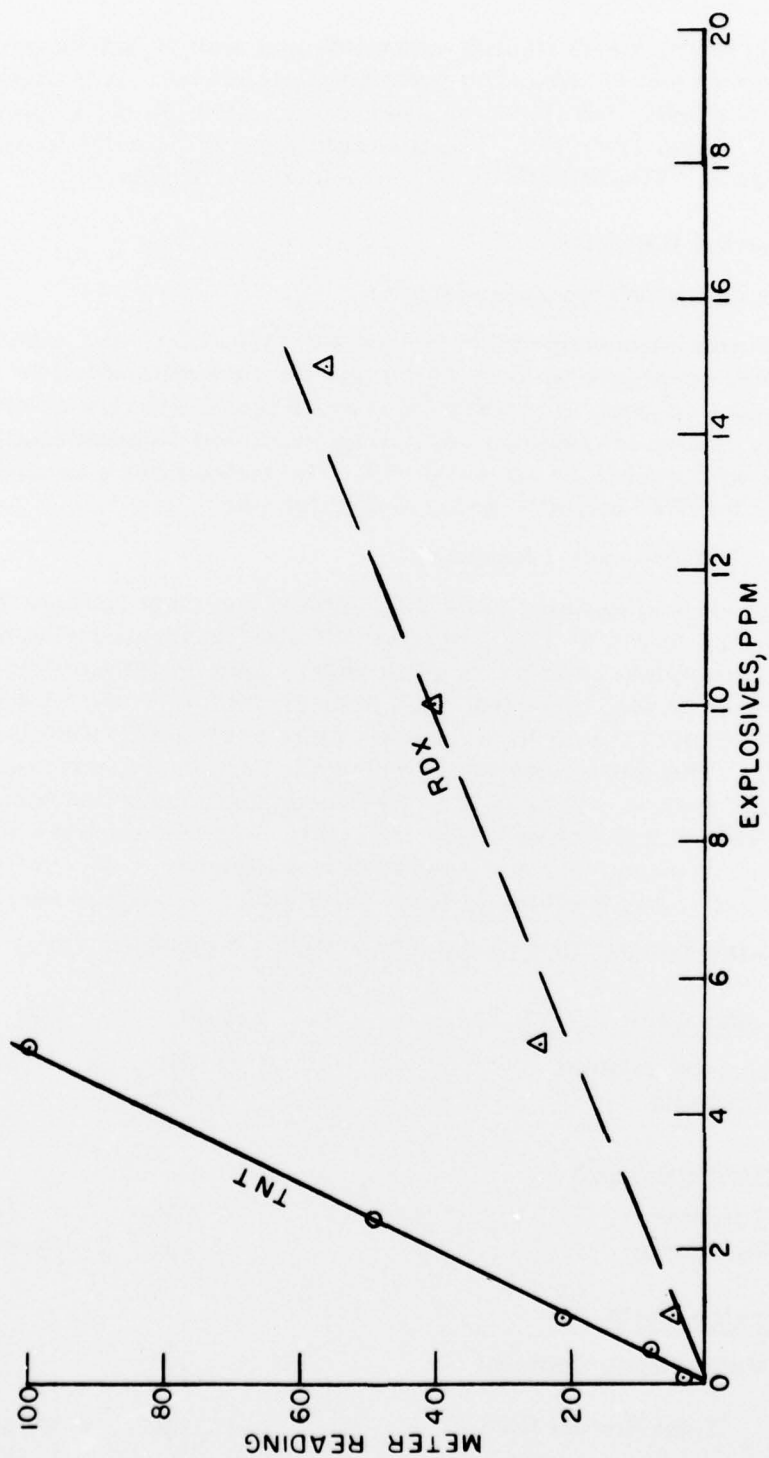


Figure 8. TNT and RDX calibration curves

Ruchhoft-Meckler Method (ref. 10)

This method uses sodium hydroxide and sodium sulfite for color development and a measuring wavelength of 505 nm. It is one of the earliest methods, but is rarely used to-day although it has been updated by S. S. Mudri (ref. 11). The disadvantages are similar to those of the Silas Mason. The sensitivity of the method is 2.0 ppm.

Instrumental Methods

Liquid Chromatographic Method

A liquid chromatographic method for TNT, RDX, and tetryl in wastewater was developed at Iowa AAP using an ultraviolet detector and a Corasil C₁₈ column. This method is much faster than the colorimetric methods and several explosives can be identified simultaneously. Operating conditions are listed in table 4. The method has a sensitivity of 0.4 ppm for TNT and RDX and 2.0 ppm for tetryl.

Spectrophotometric Method

This method appears to be the simplest and most reliable for continuous monitoring of TNT and other nitrocompounds in wastewaters. In the carbon adsorption process for pink water, Iowa AAP found that RDX appears in the eluate several days prior to TNT (ref. 5). This order of elution was also shown in an earlier, more systematic study by Vlhakis (ref. 12). Therefore, when monitoring the carbon columns, note that the first signal is due to RDX. The concentration reaches about 15 ppm of RDX before the TNT emerges (ref. 12). The DuPont 400 photometric analyzer, at measuring and reference wavelengths of 280 and 365 nm, respectively, has been found to be suitable for this application.

MONITORING COSTS (AUTOMATION VS LABORATORY)

<u>Capital Costs</u>	<u>Auto</u>	<u>Lab</u>
Instrumentation	\$ 10,000	\$ 2,500
Recorder	1,000	
<u>Recurring Costs</u>		
Supplies	100	750
Personnel	5,000	50,000
<u>Service Costs</u>		
Maintenance Contract	400	250
Total Annual Cost	\$ 16,500	\$ 53,500

Table 4. Liquid chromatographic conditions for analysis of explosives in water

Instrument:	Waters associated ALC 202		
Column:	Corasil C ₁₈ , 2 ft x 2.3 mm I.D.		
Solvent:	Water: Acetonitrile (2: 1)		
Flow rate:	0.9 ml/min (30% pump stroke)		
Recorder:	Leeds & Northrup Speedomax H		
Chart speed:	60 cm/hr		
Sample size/range:	TNT and tetryl	2-40 ppm, 10μl/08	
	TNT	1-10 ppm, 10μl/04	
	TNT	0.4-5 ppm, 20μl/04	
	RDX	0.5-20 ppm, 10μl/04	
	RDX	0.2-5 ppm, 20μl/04	
Elution times:	TNT	3.5 minutes	
	RDX	2.1 minutes	
	Tetryl	1.5 minutes	

Based on these data, on an 8-hr/day basis, the cost ratio of automation vs laboratory analysis is about 1:3, or a savings of \$37,000/yr. Should the operation be on a 24-hr/day basis, the savings would be proportionately greater.

CONCLUSIONS

1. The DuPont analyzer has proven to be a suitable instrument for the continuous monitoring of TNT and/or RDX by ultraviolet absorption.
2. The method follows Beer's Law at all concentrations of TNT and RDX.
3. The operation of the analyzer is very cost effective.

RECOMMENDATIONS

1. Longer cells and other wavelengths should be studied to determine the condition which will provide adequate sensitivity for future requirements.
2. The behavior of RDX and TNT within the carbon column should be studied with respect to breakthrough patterns.
3. AAP's should monitor the effluent from carbon columns using calibration curves derived from the method described under Operating Procedures.
4. The photometric analyzer should be used to monitor other processes being studied, or adopted, for abating pollution from pink water.

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